



ATTEMPTS AT THERMOLUMINESCENCE DATING OF FIRED MATERIALS FROM THE PRZEWORSK CULTURE SETTLEMENTS

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Abstract: The thermoluminescence (TL) dating method has a significant measurement error margin reaching almost 10%. Due to this fact it could be considered as little effective in case of such sites from the Roman period as burial grounds with many artefacts useful for archaeological dating. However, for many settlements from this period, where pottery is the only kind of artefacts, the TL method can give notable results. The main purpose of the study was to make an attempt at TL dating of pottery and clay daub samples from the Nieszawa Kolonia and Kręcieszki sites and to compare the obtained dates with the results of archaeological dating of selected features from the Przeworsk Culture settlements. In the Kręcieszki site the fragments of burnt clay daub were dated by the TL method for the first time in the Lublin laboratory. It turned out that clay daub is an equally good dating material as pottery. It can be found that the TL dating of pottery from Nieszawa Kolonia confirms two stages of settlement. The first settlement stage is related to the phases B2-B2/C1-C1a of the Roman period, i.e. from the beginning of the 2nd to the beginning of the 3rd century. The second group of TL dates corresponds to the phases C2D that is to the second stage of settlement, from the second half of the 3rd century to the half of the 5th century AD. The results of TL dating of pottery and clay daub in the Kręcieszki site are rather similar and correspond to the phase B1/B2 of the period of Roman influence, determined from pottery style, but can also indicate the phase B2/C1.

Keywords: TL dating, pottery, clay daub, Roman period, Migration period, Przeworsk Culture.

1. INTRODUCTION

The Nieszawa Kolonia village (Opole county, Lublin province) is situated in the Małopolska Vistula River Gap belonging to the Lublin Upland (**Fig. 1**) (Kondracki, 1994).

The Przeworsk Culture settlement occurring in this village is the best examined settlement site from the older Roman period in the Lublin region, and the second after the Drążgów site, which provides information about

dwelling construction of the Przeworsk Culture population (Kokowski, 1989; Stasiak-Cyran, 2000). Its total area can reach even 1 ha though only 2215.8 m² has been discovered to date (Stasiak-Cyran, 2005; 2008). Twelve dwelling features were described in this site to date (**Fig. 2**).

The settlement in Nieszawa Kolonia, the site No. 5, does not differ from the settlement schemes found in the other areas of the Przeworsk Culture (cf. Michałowski, 2003), both in respect of its situation and pattern. It is situated on a small promontory rising over the floodplain of the Wyżnica River near its mouth to the Vistula River (**Fig. 1**). The arrangement of features is random, without

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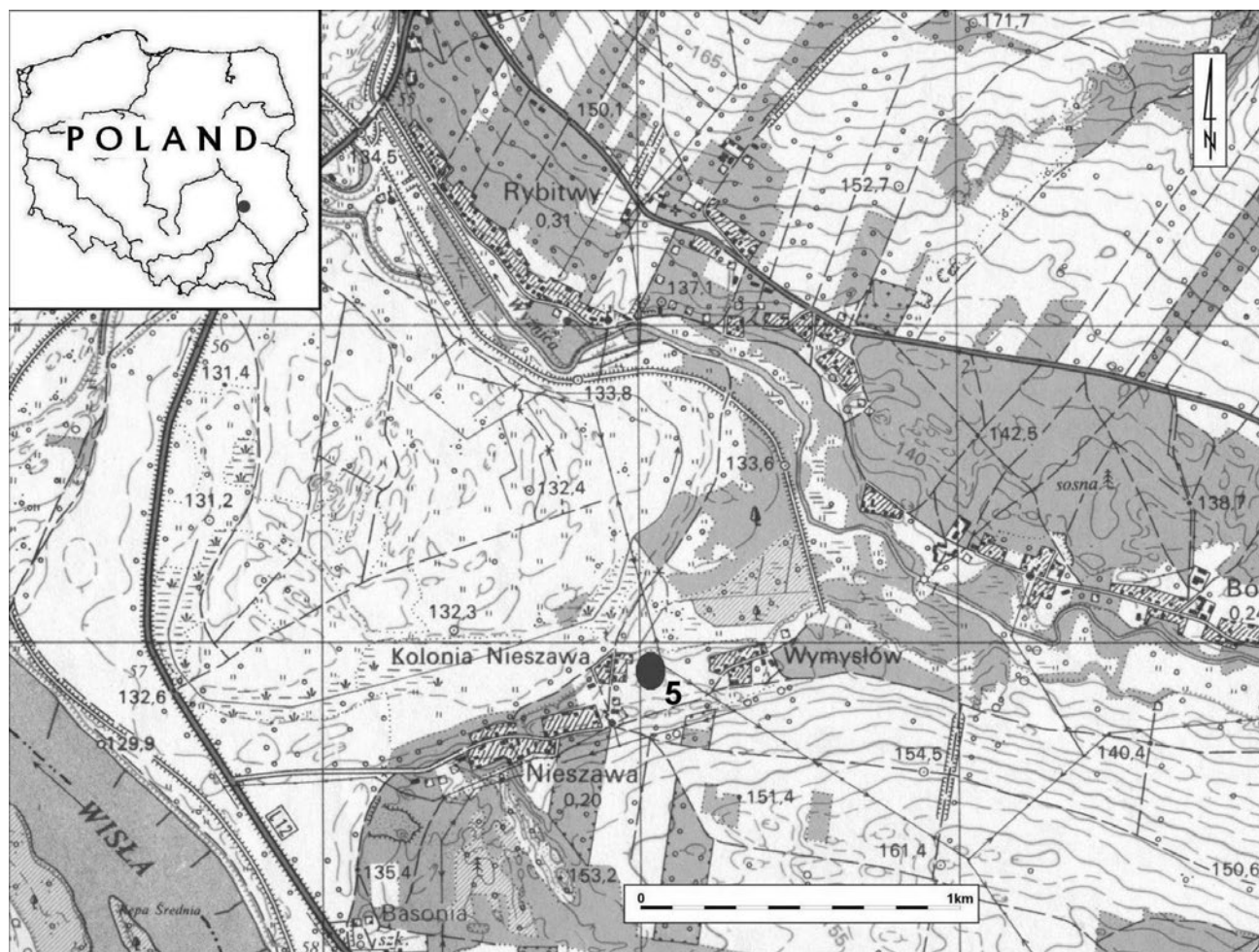


Fig. 1. Location of the Nieszawa Kolonia site

division into dwelling and production zones. It is rather a farmstead type of spatial arrangement, with dwelling structures accompanied by domestic pits of different functions and hearths. The buildings regarded as dwellings have post construction only. The floor area of “houses” is from a dozen or so to over 50 m². They are differently oriented to the directions of the world. They are sunk into the ground from 20 to 90 cm, which indicates the overground or semi-subterranean character of buildings (Abramek, 1993; Stasiak-Cyran, 2005).

Most artefacts and features found in the Nieszawa Kolonia date from the phases B2b-B2/C1-C1a, i.e. from the period of development peak of the Przeworsk Culture settlement and then its collapse because of the expansion of the Wielbark Culture population in the Lublin region (Kokowski, 1991). However, besides these materials, among the pottery fragments occurring in the fillings of pits there are the fragments of Late Roman wheel-made pottery, which have been found in the Lublin region only in the sites of the Wielbark Culture and Masłomęcz Group (cf. Kokowski, 1988). The fragments of big stor-

age vessels (so-called Krausengefässe) and metal artefacts such as bronze fibulae of VI Almgren group (late variant of A.158 brooch, two A.162 specimens and one A.166/167 specimen) seem to be exceptional findings (Stasiak-Cyran, 2000; 2005; 2008). In Silesia such storage vessels are related to the phases C3-D1 (Mączyńska, 1999). They are also known from many settlement sites from the Late Roman period in south-eastern Poland (Barłowska, 1984). Bronze fibulae are often found in the Przeworsk Culture sites from the younger Roman period (Szydłowski, 1964; Godłowski, 1981; Mączyńska, 1999).

The investigations carried out at the Nieszawa Kolonia site shed new light on the problem of the decline of the Przeworsk Culture settlement in south-eastern Poland. The occurrence of findings, dating probably from the phases C3-D1 of the Roman period, confirms that it is possible, as it has been signalled in literature (Kokowski, 1991), to discover features of the Przeworsk Culture somewhat younger than those discovered earlier. Therefore, the site was most probably settled again in the decline phase of this culture. According to the former state

of knowledge, the settlements of the Przeworsk Culture did not occur to the north-east of the San River in that time (Mączyńska, 1999). The discoveries in the site No. 5 in Nieszawa Kolonia make undoubtedly a breakthrough in the knowledge of settlement in the Lublin region in the Roman period (Stasiak-Cyran, 2000).

In 2008, based on archaeological materials obtained during the exploration, an attempt was made at TL dating of the site No. 5 in Nieszawa Kolonia. These analyses were a part of the project entitled: "Rescue investigations of the Przeworsk Culture settlement in Nieszawa Kolonia, Opole Lubelskie county: research with the use of non-invasive methods, specialist analyses, protection of the most endangered parts of the site from destruction".¹

The main purpose of the study was to make an attempt at TL dating of pottery and clay daub² samples and to compare the obtained dates with the results of archaeological dating of selected features. The next objective was to confirm the two stages of settlement: the first one during the phases B2 to B2/C1 – C1a, and the second one from the phase C2 or most probably C3 perhaps to the end of the phase D1 (?) as indicated by relative chronology determined from, among others, fragments of storage vessels (so-called *Krausengefässe*) and some metal artefacts (among others Stasiak-Cyran, 2000; 2008).

The site No. 1 in Kręcieszki was discovered in 1985 by the local farmer in Bedlno district (Kutno county, Łódź province), near the road A2 to Warsaw on low sandy elevation (Fig. 3). The site is situated on the Kutno Plain belonging to the Central Mazovian Lowland (Kon-dracki, 1994).

The excavations in the site No. 1 were conducted in five field seasons in the years 1987-1988 and 2005-2007. The site, at first considered as the monocultural burial ground of the Pomeranian Culture population with cist graves dated to the Hallstatt D and early La Tène periods, turned out to be the multicultural site.

The investigations revealed three settlement stages in this site. The first stage was connected with the Lusatian Culture (Rychter, 2010). The following units belonging to the settlement of the Lusatian population were found: storage pits, hearths, and probably remains of dwelling buildings (Rychter, 2010). Numerous fragments of pottery and flint material were dated to the IV/V period of the Bronze Age.

Then the area was settled by the Pomeranian Culture population (Rychter and Stasiak, 2006). Three cist graves discovered in the 1980s, and three urn burials beneath larger inverted urns found in 2007 were related to this culture.

The last settlement stage was that of the Przeworsk Culture population. The following traces of the Przeworsk Culture were found during the excavations conducted in 2005: one fragment of clay daub, loom-weight, and few fragments of pottery preliminary dated to the phase B1/B2 of the Roman period. The next remains of the settlement were found in the years 2006-2007. The part of dwelling feature discovered in 2006 measured four by two metres. It was very compact, burnt layer of clay daub about 20 cm thick. In the field season 2007 the second part of this feature was found, i.e. its south-eastern corner. This relic measured 2.1 by 0.7 metres and was composed of two clay daub layers with the total thickness of about 25 cm. Thus, this big building measured approximately 4.7 by 4.1 metres. None post holes were found so perhaps frame construction was used in buildings in this site.

The relic, probably of a small cellar with a diameter of about 50 cm and depth of 30 cm, was found under the north-western corner of the house. It was strongly burnt clay daub with the fragments of pottery and lime. The pottery and clay daub were selected for TL dating.

2. DESCRIPTION OF DATING METHOD

In the case of pottery or clay daub, the TL dating determines the time which has passed since it was burnt. The TL age is a quotient of archaeological dose (AD) and dose rate (DR). The archaeological dose is a sum of the equivalent dose (ED) and correction (I) due to the fact of non-linear increase of thermoluminescence for small absorbed doses (Mejdahl and Wintle, 1984).

The determination of TL age of pottery and clay daub proceeds as follows. The dose rate is calculated from the measured concentrations of natural radioisotopes in a sample of pottery and sample of deposit in which it was found. The concentrations of radioisotopes obtained in Bq/kg are converted into absorbed dose rates for α , β and γ radiation in Gy/ka, based on the data published by Adamiec and Aitken (1998). In the calculation of DR the correction ($DR_c = 0.15$ Gy/ka) for cosmic radiation (Prescott and Hutton, 1994) and the correction $k = 0.1$ (for small effectiveness of generating thermoluminescence when subjected to α radiation) are taken (cf. Benea *et al.*, 2007). For the samples of pottery from the Nieszawa Kolonia and Kręcieszki sites the dose rate DR (Table 1) was calculated as a sum of components coming from α and β radiation emitted by nuclides occurring in pottery sample and absorbed by mineral grains of this sample, γ radiation emitted by isotopes occurring in deposit but absorbed by mineral grains of pottery sample, and cosmic

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² **Daub:** clay which is smeared onto a structure of timber or wattle (interwoven twigs) as a finish to the surface. It is normally added to both faces of a wall and is used to keep out drafts and give a smooth finish. The material usually survives only when baked or fire-hardened, as would be the case if a structure burned down. It can usually be recognized by the impressions of the wattle to be found on its inner face (Kipfer, 2007).

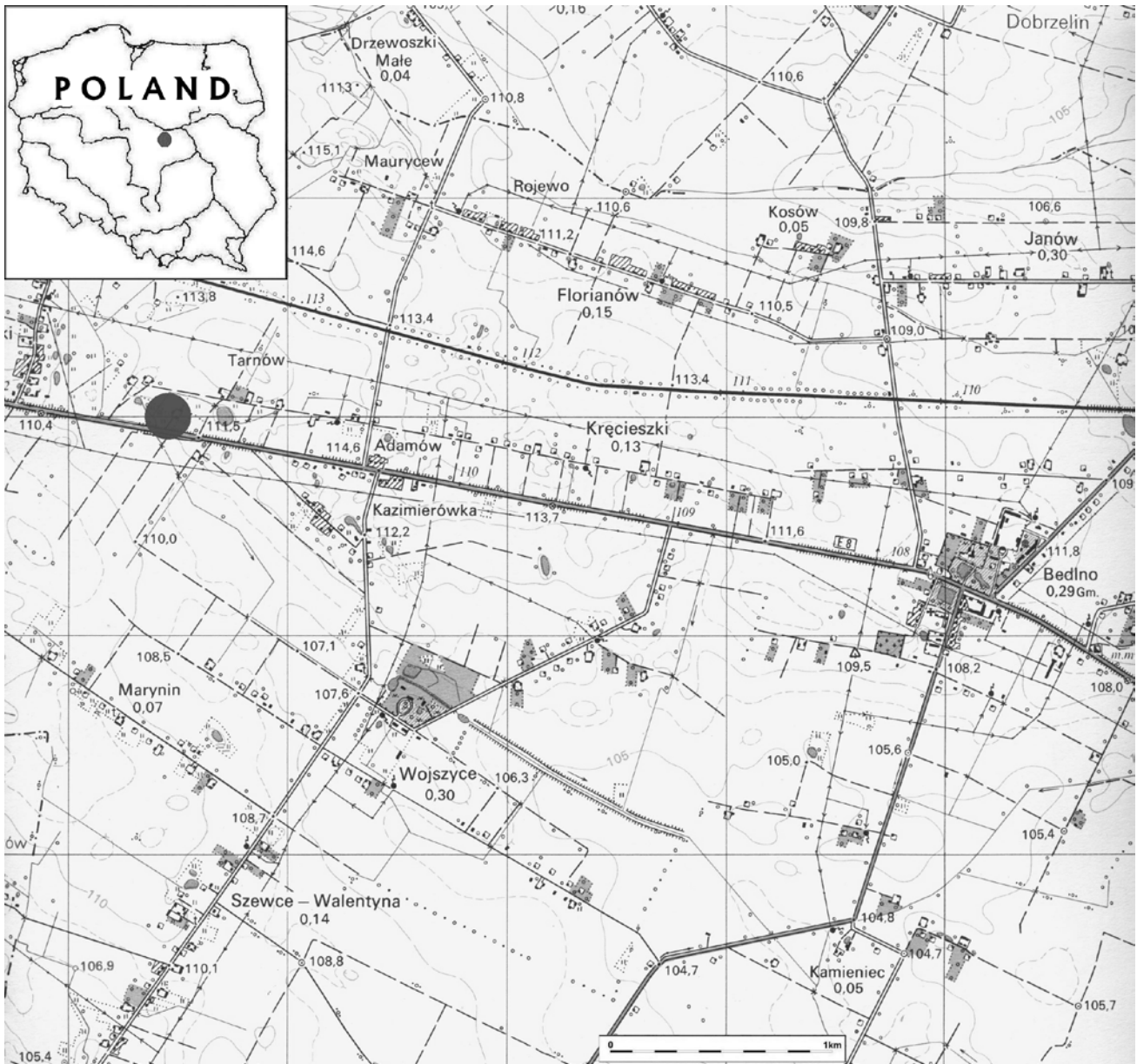


Fig. 3. Location of the Kręcieski site

radiation (Bluszcz and Adamiec, 1994; Bluszcz and Pazdur, 1994).

The measurements of radioisotopes were carried out in the laboratory using a three-channel, stationary gamma spectrometer type MAZAR-95 produced by POLONIZOT (Poland) with scintillation probe with NaI/Tl crystal. The deposit portions of about 600 g in weight in the Marinelli-type containers were put in a lead housing with 5 cm thick walls. The pottery or clay daub plates (not crushed) of 60-100 g in weight and about 60 mm in diameter were put in a specially designed lead chamber with 10 cm thick walls. The measurement time was about 70 hours (3 cycles of 80 000 seconds each) for deposit and about 200 hours (3 cycles of 240 000 seconds each)

for pottery or clay daub sample. So long time was used in order to diminish the measurement uncertainty, and the differences in the measurement time between deposit and artefact resulted from their volume.

The DR was calculated with the correction for sample moisture of about 15%. Natural moisture of artefact and deposit was measured in laboratory before TL age determination. Pottery and samples of deposits were put in plastic bags in order to protect them from loss of moisture. Seasonal and long-term changes of moisture were taken into consideration basing on the data collected during geological survey carried out in the Lublin Upland for the Detailed Geological Map of Poland 1 : 50 000 (Rzechowski, 1995).

In order to determine archaeological dose a pottery or clay daub sample of about 10 g in weight was taken and its 2-3 mm thick external layer was removed. Then a sample was carefully crushed and the 30-40 μm poly-mineral fraction was separated using sieve method. The obtained mineral material was cleaned by etching with acetic acid (1:1) for 30 minutes (Martini *et al.*, 2001). Feldspars constitute only several per cent (determined by staining of feldspar) of this fraction so separation of minerals was not made. The mineral material was divided into two parts. The first one was divided into several portions. One was left as natural, and the remaining por-

tions were irradiated with the ionising radiation doses of 2.5 Gy, 5 Gy, 7.5 Gy and 10 Gy. Then the mineral material was divided into the portions of 4 mg in weight, which were placed in the TL reader/analyser RA'94 type (with the EMI 9789 QA photomultiplier) produced by Mikrolab Kraków (Poland). Aliquots are glow out in argon atmosphere at a heating rate of 10°C/s up 400°C. Based on the obtained glow curves (relationship between thermoluminescence and temperature, Fig. 4), the equivalent dose (ED) was determined by additive method (Fig. 5A). In order to determine the correction (I) due to the fact of non-linear relation $TL = f(D)$ for small absorbed

Table 1. The values of dose rate (DR), archaeological dose (AD) and TL age of pottery and clay daub.

Lab. No. of sample Lub-	Inventory No.	K (Bq/kg)	U (Bq/kg)	Th (Bq/kg)	Dose rate DR (Gy/ka)	Archaeological dose AD (Gy)	TL age (a)
Nieszawa Kolonia site							
4603	4699	369.7±11.1	27.1±1.9	40.0±2.2	3.86±0.13	6.2±0.4	1610±120
4604	4727	375.5±11.3	23.9±1.7	40.9±2.4	3.62±0.17	6.4±0.4	1740±130
4605	4177	438.7±13.2	22.9±1.6	37.5±2.1	4.27±0.11	3.4±0.2	796±60
4606	4679	462.6±13.9	36.1±2.5	51.5±2.8	4.94±0.16	4.5±0.3	910±70
4607	3176	425.0±12.8	20.5±1.4	33.6±1.8	4.04±0.14	6.0±0.4	1490±110
4608	3160	412.9±12.4	13.6±0.9	41.6±2.3	4.02±0.11	6.1±0.4	1520±120
4609	4723	369.5±11.5	16.6±1.2	36.8±2.0	3.52±0.12	5.9±0.4	1680±130
4610	2555	383.0±11.5	22.2±1.6	26.5±1.5	3.51±0.11	8.0±0.6	2250±170
4611	4741	367.0±11.0	26.8±1.9	41.9±2.3	3.62±0.12	6.2±0.4	1710±130
4612	4434	370.0±10.9	21.6±1.5	39.5±2.2	3.48±0.13	6.5±0.5	1870±140
4613	4613	296.8±8.9	15.3±1.1	39.7±2.3	2.74±0.12	4.4±0.3	1610±150
deposit	-----	160.5±4.8	5.2±0.4	1.4±0.1	0.71±0.03	-----	-----
Kręcieski site							
4387	clay daub vessel	380.0±11.4	22.3±1.6	25.7±1.4	2.71±0.14	4.8±0.2	1780±130
4388	-----	471.8±14.2	43.4±3.0	23.5±1.3	4.99±0.23	8.5±0.4	1710±120
deposit	-----	95.7±2.9	6.3±0.4	3.1±0.2	0.57±0.03	-----	-----

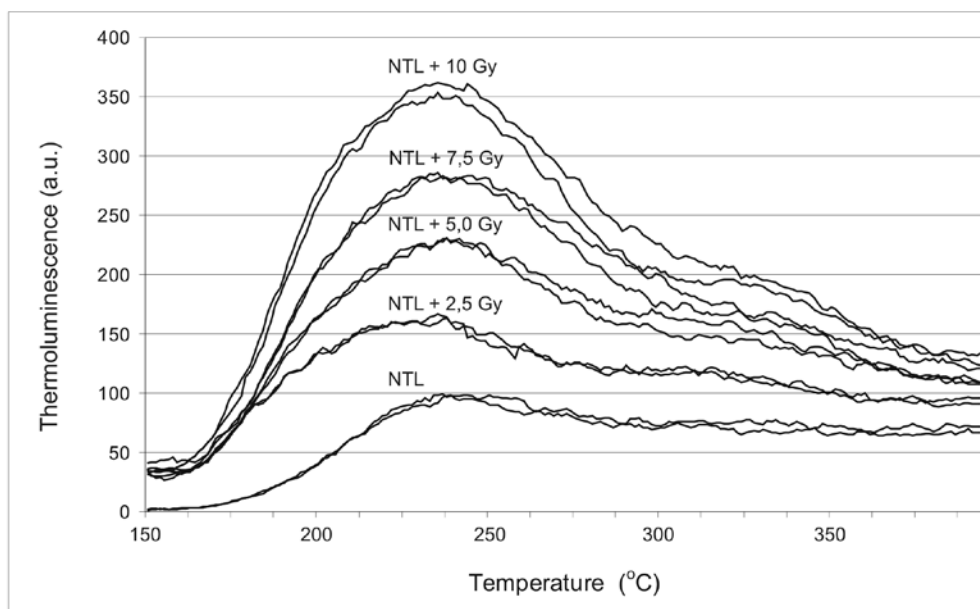


Fig. 4. Thermoluminescence glow curve for sample Lub-4606; NTL – thermoluminescence of non-irradiated part of the mineral material; NTL+2.5 Gy, NTL+5 Gy, NTL+7.5 Gy, NTL+10 Gy – thermoluminescence of parts of the mineral material irradiated with additional doses of ionizing radiation.

doses, the second part of mineral material was heated at 500°C for 3 hours. Then it was divided into portions and irradiated with the same doses as those used for ED determination (Fig. 5B). During thermoluminescence measurements an optical filter HA-3 was used to cut infra red radiation of heater. The values of dose rate (DR), archaeological dose (AD) and TL age are listed in Table 1. The TL light sum was read under narrow region of the TL glow curve, which includes its maximum (e.g. 235-245°C for the sample Lub-4606; Fig. 4). Confidence interval of the obtained values of TL age is 68%.

3. CRITERIA OF MATERIAL SELECTION FOR TL DATING

All units selected for TL dating are connected with the Przeworsk Culture. Their ages, determined using archaeological methods, fall within the above-mentioned settlement stages. There were selected the features of different function such as storage pits, dwelling buildings, hearths, post holes.

Among the selected features from the Nieszawa Kolonia site, besides pottery, other non-ceramic dating artefacts were found in seven of them (51, 59, 63, 99, 113 and 113t, 116). They enable us to assign the structures to the first or second stage of settlement functioning. Two pits containing only pottery material can be also related to the second stage because the fragments of wheel-made vessels (No. 122) and *Krausengefässe* storage vessels (No. 87) were found in their fillings. In two features

(116A and 117), from which pottery samples were taken for TL analysis, dating artefacts were absent. Therefore, they were generally related to the Roman period and early stage of the Migration period (Table 3).

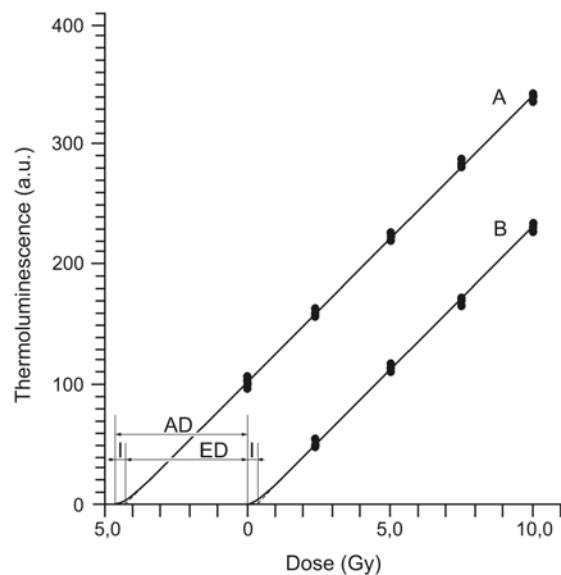


Fig. 5. Thermoluminescence growth curves for sample Lub-4606. Calculation: archaeological dose (AD) as sum of equivalent dose (ED) and correction for supralinearity (I).

Table 2. Nieszawa Kolonia, site No. 5. List of pottery and clay daub fragments selected for analysis.

Lab. No. of sample	Inventory No.	Excava-tion	Object	Object function	Description of pottery fragments selected for analysis
Lub-4603	4699	10	117	hearth	fragment of belly of thick-walled vessel of I3 group ^[1]
Lub-4604	4727	10	122	hearth	rim part of wheel-made vase-like vessel of I group ^[1]
Lub-4605	4177	8	87	storage pit	fragment of belly of storage vessel, so-called <i>Krausen-gefässe</i> , wheel-made, IIIa
Lub-4606	4679	10	116	hearth	fragment of belly of thick-walled vessel of I3 group
Lub-4607	3176	5	59	dwelling feature of post construction	fragment of belly of thick-walled vessel of I3-I4 group
Lub-4608	3160	5	63	storage pit	fragment of belly of thick-walled storage vessel of I2-I3 group
Lub-4609	4723	10	116 A	storage pit	base part of small average-walled vessel of I2 group
Lub-4610	2555	4	51	dwelling feature of post construction	rim part of thin-walled vase-like vessel of II2 group
Lub-4611	4741	10	113	farm building (?) of post construction	fragment of belly of thick-walled storage (?) vessel of I3 group
Lub-4612	4434	9	99	hearth	fragment of belly of thick-walled vessel of I3 group
Lub-4613	4613	10	113 „t”	post hole	fragment of clay daub with imprints of wood from building construction

¹Symbols denoting technological groups and subgroups are adopted from the study of hand-made pottery of the Przeworsk Culture from the Lublin region, based on the material from the burial ground in Opoka, Puławy county. These are the following groups: pottery with blackened surface // and brown pottery III, and within them: polished /I/, burnished /2/, coarse /3/, roughened /4/, coarse, in places burnished /2-3/, coarse, in places roughened /3-4/ (M. Stasiak 1994, p. 43-45). The mentioned paper contains the detailed description of the technological features of these units.

²Wheel-made vessels of the Przeworsk Culture are described according to the technological classification of the materials from Igołomia, Cracow county. These are the following groups: pottery with coarse surface, so-called kitchen //, pottery with burnished surface, so-called table III/, storage vessels of coarse surface IIII/ (H. Dobrzańska 1990, p. 16-20). The mentioned paper contains the further detailed description of the technological features of these units.

Table 3. Nieszawa Kolonia, site No. 5. List of the results of TL dating of pottery and clay daub.

Lab. No. of sample	Inventory No.	Relative chronology of objects (AD)	TL age (BP)	TL age (AD)
Lub-4603	4699	OR- OWL ¹ (about 0 AD - about 450 AD)	1606±122	402±122
Lub-4604	4727	phases C2 - D (about 260 AD - about 450 AD)	1740±132	268±132
Lub-4605	4177	phases C2 - D (about 260 AD - about 450 AD)	796±60	1212±60
Lub-4606	4679	phases B2/C1 - C1a (about 150 AD - about 210-230 AD)	911±69	1097±69
Lub-4607	3176	phases B2/C1 - C1a (about 150 AD - about 210-230 AD)	1485±113	523±113
Lub-4608	3160	phases C3 - D (about 310 r. - about 450 AD)	1517±115	491±115
Lub-4609	4723	OR - OWL (about 0 AD - about 450 AD)	1676±127	332±127
Lub-4610	2555	phases B2/C1 - C1a (about 150 AD - about 210-230 AD)	2251±171	243±171 BC
Lub-4611	4741	phases C2 - D (about 260 AD - about 450 AD)	1713±130	295±130
Lub-4612	4434	phase B2 (about 80 AD - about 150 AD)	1868±142	140±142
Lub-4613	4613	phases C2 - D (about 260 AD - about 450 AD)	1606±145	402±145

¹OR – Roman period (period of Roman influence), OWL – Migration period

The pottery samples were selected taking into account also their representative character in respect of their technological and morphological features typical of the vessels of the Przeworsk Culture from the Roman period. The detailed description of the pottery fragments selected for analysis is presented in **Table 2**. For the groups and types of hand-made pottery it is based on the materials from the burial ground in Opoka, Puławy county (Stasiak, 1994) and for wheel-made pottery – on the classification of vessels from Igołomia, Cracow county (Dobrzańska, 1990).

In the Kręcieszki site, besides pottery, dating artefacts were absent. Moreover, pottery is not very typical. In order to check the TL age obtained for pottery, we decided to make an attempt at TL dating of other materials. Therefore, for the first time in the Lublin laboratory, a fragment of burnt clay daub was TL dated (Kusiak *et al.*, 2009). TL dating of burnt house daub (12 samples from Birini Site in Northern Ghana) was described by Godfrey-Smith *et al.* (1997). The obtained results correspond well to the radiocarbon ages suggesting that reliable TL dating results can be obtained for burnt house daub.

4. RESULTS AND DISCUSSION

In case of the Nieszawa Kolonia site, eight of eleven obtained TL results are within the time-frame of the Roman and Migration periods, i.e. between the 1st and 5th century AD (**Table 3**). Five of these TL ages, obtained for clay daub and pottery, well correspond to the stages of

settlement functioning determined from dating artefacts. One TL dating result is incompatible with the detailed relative chronology within the Roman and Migration periods. (**Table 3**, inventory No. 3176). Two TL ages obtained for pottery fragments generally dated to the Roman and Migration periods (**Table 3**, inventory No. 4723 and 4699) could be also regarded as positive results.

We can hazard a guess that the TL dating of pottery and clay daub from the Nieszawa Kolonia site confirms two stages of settlement. One TL age (140±142 AD) corresponds to the first stage of settlement functioning, i.e. the phase B2b of the Roman period. As many as six TL ages, ranging from 268±132 to 491±115 AD, correspond to the phases C2 – D, i.e. to the second stage of settlement, from the second half of the 3rd century to the half of the 5th century AD.

Three dates obtained by the TL method are completely incompatible with the accepted chronology of the features from which the samples were collected. Two TL results indicate the early Middle Ages (**Table 3**, inventory No. 4679 and 4177) and one – the early pre-Roman period (**Table 3**, inventory No. 2555). It should be stressed that faulty dating of these samples using archaeological methods is very little probable. The selected samples are fragments of typical (in respect of technology and form) vessels from the Roman period. The incompatibility of the TL date with the result of archaeological dating is especially disappointing in case of the sample of inventory No. 4177 from the pit 87 as it is the fragment of belly of thick-walled storage vessel with everted rim,

so-called *Krausengefässe*, dated to the phase C2-D of the Roman period (Stasiak-Cyran, 2008).

In the Kręcieszki site the results of TL dating of pottery and clay daub are rather similar and correspond to the phase B1/B2 of the period of Roman influence, determined from pottery style, but can also indicate the phase B2/C1 (Table 1). The TL dating of burnt clay daub was successful. It turned out that clay daub is equally good dating material as pottery.

5. FINAL REMARKS AND CONCLUSIONS

It should be stated that thermoluminescence dating of artefacts from the Nieszawa Kolonia and Kręcieszki sites was successful.

The main value of the TL analysis carried out for the Nieszawa Kolonia site is the fact that quite many fragments of pottery were dated. The results confirm the chronology deduced from relative dating materials, and the obtained TL ages indicate age brackets of two settlement stages. However, we do not find any chronological order of the TL ages of pottery representing different technological groups and styles. Many more TL dating results should be obtained to observe possible relations between the occurrence of pottery representing different technological groups and styles and particular phases of the second settlement stage.

It is also important that TL dating enables us to determine the chronology of features without good relative dating materials. Such a situation occurred both in the Nieszawa Kolonia and Kręcieszki sites.

It is advisable to carry out TL dating of at least several fragments of pottery collected in one site in order to minimize the possibility of false determination of the site age. Interpretation of single results can lead to wrong conclusions. Such a situation we have in the Nieszawa Kolonia site. Single fragments of pottery, the TL age of which was incompatible with the expected age, occurred also in almost all series of samples from several sites dated in the Lublin laboratory. Many TL dating results obtained for one feature give the possibility to separate accidental results from those well dating the examined structure. In this stage of the investigations we cannot identify the causes of this incompatibility. The causes of the above mentioned incompatibility are an important problem to solve.

Pottery is commonly used in the TL dating of archaeological features (Preusser *et al.*, 2008; Wintle, 2008). However, burnt clay daub is also good dating material, as it was evidenced by the obtained TL ages.

The obtained results enable us to make good predictions for future TL dating of archaeological fired materials.

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